

This Page Is Inserted by IFW Operations
and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

**As rescanning documents *will not* correct images,
please do not report the images to the
Image Problem Mailbox.**

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
12 September 2003 (12.09.2003)

PCT

(10) International Publication Number
WO 03/074650 A1

- (51) International Patent Classification⁷: C12G 3/08,
C12H 1/14, 1/16, B01D 61/58
- (21) International Application Number: PCT/IT02/00139
- (22) International Filing Date: 5 March 2002 (05.03.2002)
- (25) Filing Language: Italian
- (26) Publication Language: English
- (71) Applicant (for all designated States except US): VELO SPA [IT/IT]; Via Piave, 55, I-31030 Altivole (IT).
- (72) Inventors; and
- (75) Inventors/Applicants (for US only): FATUTTO, Daniele [IT/IT]; Via Riviera Marco Polo 47, I-30174 Venezia (IT). SALVADOR, Sergio [IT/IT]; Via Carlo Forlanini '47, I-31029 Vittorio Veneto (IT). VELO, Antonio [IT/IT]; Via Madonette, 39, I-31031 Caerano San Marco (IT).
- (74) Agent: AGOSTINI, Agostino; Dragotti & Associati S.r.l., Via Paris Bordone, 9, I-31100 Treviso (IT).
- (81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU,
- (84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Declaration under Rule 4.17:

— of inventorship (Rule 4.17(iv)) for US only

Published:

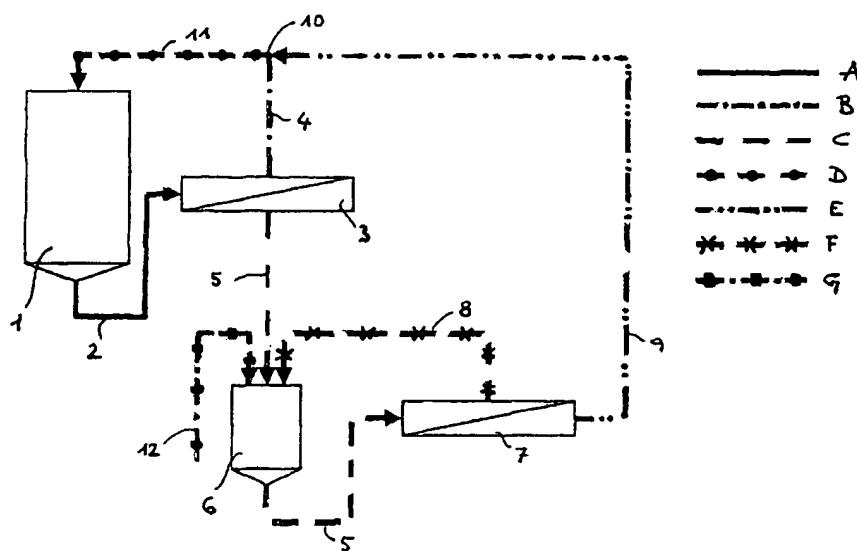
— with international search report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: PROCESS AND APPARATUS FOR WINE TREATMENT TO REDUCE ITS CONTENTS OF VOLATILE ACIDITY



WO 03/074650 A1



(57) Abstract: For deacidifying wines, i.e. reducing the volatile acid compounds that are present as a solution in the wine, the present invention provides two successive process stages. In the first one of such stages, the solution to be processed (A) is separated into a first concentrate (B) and a first permeate (C), in which the latter comprises said compounds. In the second one of such stages, said first permeate (C) is in turn separated into a second concentrate (F), in which there are included the compounds that are responsible for said volatile acidity, and a second permeate (E) that comprises the solvent. Preferably, said first permeate (C) is at least partially neutralized with high-pH additives between said first and said second stage.

5

10 **PROCESS AND APPARATUS FOR WINE TREATMENT TO
REDUCE ITS CONTENTS OF VOLATILE ACIDITY**

DESCRIPTION

15 The present invention refers to a process and an apparatus aimed at deacidifying wines, i.e. reducing the contents of volatile acid compounds thereof.

20 Organoleptic qualities and characteristics of wines in general are largely known to be adversely affected by volatile acidity, i.e. the amount of volatile acid compounds, among which acetic acid and ethyl acetate, which are present as a solution in wines, are of particular purport in this connection.

25 Fully understandable is therefore the effort being made by wine producers in view of achieving a reduction in the volatile acidity of wine, or even eliminating such acidity altogether, to the purpose of optimising the overall quality of theirs products. The use of such products as calcium carbonate, potassium bicarbonate and neutral potassium tartrate directly in the wine in order to achieve a deacidification thereof fails to be effective as far as acetic acid is concerned, since neutralization of weaker acids only,
30 such as in particular tartaric acid, is obtained in this way.

The sole possibility that really exists to effectively reduce the volatile acidity of a wine lies in blending such a wine with another one having a very low volatile acidity.

It therefore is a main purpose of the present invention to provide a selective method, i.e. a method that is capable of solely reducing the volatile acidity, in such a manner as to avoid affecting other compounds, especially acid ones, which on the contrary favourably contribute to the final quality of the wine, and furthermore to avoid blending of different wines.

10 A further purpose of the present invention is to reduce the usage of additives required to this aim, as well as reduce the physical and economic dimensions of the equipment used to reduce the volatile acidity of wine, so as to make it also available to small producers, who usually cannot rely on the availability of huge economic resources, and/or in view of being able to 15 use the invention for processing wines that may be termed as niche products from a marketing viewpoint.

According to the present invention, these and further aims are reached in a process and an apparatus embodying the characteristics as recited in the appended claims.

20 The features and advantages of the present invention will be anyway more readily understood from the description of a preferred, although not sole embodiment that is given below by way of non-limiting example with reference to the accompanying drawings, in which:

25 - Figure 1 shows a diagrammatical view of a continuous separation process for deacidifying wines and also bears the legend of the symbols used to indicate the various media involved in the process as mentioned in this description;

- Figure 2 is a schematic view of the layout of a continuous separation apparatus;

30 - Figure 3 is a view similar to the one shown in Figure 1, however

relating to a discontinuous separation process;

- Figure 4 is a simplified view of the main section of a portion of the apparatus shown in Figure 2, where a treatment takes place intended to neutralize the volatile acid substances.

With reference to Figure 1, the product to be treated - which is generally constituted by wine, but will be referred to as solution A hereinafter since it contains, in form of solutes, the volatile acid compounds to be eliminated - is at the beginning of the process in a storage area 1, namely an initial reservoir, from which it is then transferred via a first delivery line 2 to a process station 3 where a first separation stage takes place. Said station 3, which is also herein referred to as the initial station, comprises at least a membrane which, depending on the size of the molecules of the substances to be removed from the solution A, may either be a reverse-osmosis membrane (i.e. adapted to retain molecules with a size less than 200 dalton), or a nanofiltration membrane (i.e. suitable for molecules with a size of 200 to 1,000 dalton) or a ultrafiltration membrane (i.e. suitable for molecules with a size of 1,000 and 300,000 dalton). In this manner, the solution A is separated into two parts that will be referred to herein as the first concentrate B, which contains most of the solutes that have desirably to be retained, and the first permeate C, which contains the solutes that have on the contrary to eliminated, further to the solvent and a small proportion of the solutes that have desirably to be retained.

Said first concentrate B is let into a first connection line 4 so as to return to the storage area 1 for the reasons that shall be explained to greater detail further on. The first permeate C is in turn transferred, via a second delivery line 5, up to a new process station 7, which is also herein referred to as the final process station, where a second separation process takes place that is much similar to the first one, but is performed under utilization of more selective membrane means so as to be able to obtain, starting from said first permeate C, a second permeate E containing the

totality, or almost the totality, of the solvent and the solutes to be retained in the product, and a second concentrate F containing the undesired compounds, i.e. the compounds that have on the contrary to be removed from the final product.

5 Said second permeate E is delivered via a second connection line 9 to a connection point 10 in view of being caused to mix with the first concentrate B so as to give rise a so-called reconstituted wine D, which is then caused to flow back into the initial reservoir 1 via a return line 11 to again go through the above described process steps until the desired result
10 is eventually achieved.

The second concentrate F is in turn delivered back to the final process station 7. Somewhere along the second delivery line 5 there may be included a reaction stage 6 to which is fed not only with the first permeate C, but also with the second concentrate F and one or more additives with a
15 high pH value, that may be present either in a liquid or a solid form, via a third delivery line 8 and a fourth delivery line 12, respectively. In the case that solid additives are used, said fourth delivery line 12 shown in figure 1 shall of course not be intended as being a pipe, but rather as an indication of the use of said additives in this stage of the process.

20 In this connection, Figure 4 is shown to illustrate an embodiment of the reaction stage 6, which turns out as being particularly advantageous exactly when solid additives, such as calcium carbonate, are used in the process. The solid additive (or additives, as the case may be) G is filled into an upright container 61, which has a grille 62 as a bottom wall, and which is
25 housed within a tank 63. The first permeate C reaches the tank 63 via the inflow branch 55 of the second delivery line 5, and is caused to percolate through the additive G in such a manner as to release the volatile acid compounds contained therein. Thereupon, downstream of said bottom grille 63, it flows into the outflow branch 56 of the same delivery line 5 after
30 having first moved through a siphon 64.

Along the delivery line 8, the second concentrate F is delivered back to said reaction stage 6, which in this way is adapted to also act as a reservoir supplying the final process station 7.

From the description given above, the peculiar advantages of the
5 present invention can be most clearly inferred, i.e.:

- the present invention makes use of just a limited quantity of additives, since the product to be processed solely and only consists of the first permeate C, and therefore is not the totality of the wine. This in turn permits the use of smaller sized equipment and clearly contributes to keep
10 at a low level the production cost of the wines;

- there is no risk for also tartaric acid - and other compounds that favourably contribute to the quality of the wine - to be eliminated at the same time as said volatile acid compounds.

The process according to the present invention can be carried out
15 using a continuous apparatus, the layout of which is illustrated in Figure 2 where the same reference numerals are used as the ones used in the functional flow-diagram of Figure 1, whenever possible. This layout also illustrates some so-to-say ancillary equipment which is really neither strictly necessary nor sufficient in view of characterizing the present
20 invention, and for which all those skilled in the art may well be able to devise and find out a different practical implementation. Further to various pressure measurement devices, which are not shown for reasons of greater simplicity, the ancillary equipment shown in the layout of Figure 2 includes:

25 - along the first delivery line 2 (which feeds the solution A to be processed), a supply valve 21, a first pump 22, a flow-rate adjustment valve 23, a safety prefilter 25, and a second pump 26. Said safety prefilter 25, which is in turn provided with a bleeder valve 28, is used to prevent any damage to the membranes that are used as a part of the two process
30 stations 3, 7 by possible solid particles present in the solution A;

- along the first connection line 4 (in which, downstream of the initial process station 3, where the first separation stage takes place, there flows the first concentrate B so as to reach the connection point, which is not shown here for reasons of greater simplicity, with the second permeate E), a flow-rate meter 41 and a pressure adjustment valve 42;

5 - along the second delivery line 5 (in which there flows the first permeate C, and which is shown in this preferred embodiment to pass through the reaction column 6), a flow-rate meter 51 and a first adjustment valve 52 ahead of the inlet 55 into the reaction column 6, a second adjustment valve 53 and a third delivery pump 54 between the outlet 56 of the reaction column 6 and the inlet of the final process station 7, where the second separation stage is carried out;

10 - along the third delivery line 8 (along which there flows, from the final process station 7, the second concentrate F to reach the reaction column 6), a flow-rate meter 81 and a pressure adjustment valve 82;

15 - along the second connection line 9 (in which there flows the second permeate E before mixing up with the first concentrate B at the above mentioned connection point which, as already pointed out, is not shown in Figure 2), a flow-rate meter 91 and a valve 92;

20 - along the fourth delivery line 12, an adjustment valve for the additive or additives G prior to the inlet thereof into the reaction column 6.

In view of practically achieving the same quality results that can be reached with the use of a continuous apparatus such as the one illustrated in Figure 2, the process according to the present invention may as an alternative be carried out in a discontinuous manner in an apparatus which, as compared with the above described one, has the additional advantage of needing a lower economic investment, since a definitely smaller extent of means are used in such an apparatus, so that the latter is particularly suited for use by small-scale wine producers.

25 30 An example of a discontinuous apparatus in this sense is

schematically illustrated in Figure 3, in which the same graphical symbols are used as in the Figures illustrating a continuous apparatus to indicate the various process media. The possibility exists in this of using only one process station for carrying out both the first and the second filtration stage,
5 obviously according to a different timing and possibly under utilization of different filtration membranes.

The first separation stage which the solution A, stored in a first reservoir 100 and delivered via a first delivery line 110, is caused to undergo at the process station 200, produces the first concentrate B, which
10 then flows back into the same reservoir via the return line 120, and the first permeate C, which is in turn delivered via the connection line 130 to a second reservoir 140 where it is collected and temporarily stored until the second separation stage takes place.

The second separation stage, which is also performed in said process
15 station 200, solely concerns said second permeate C which reaches said process station 200 via the delivery line 150, after the temporarily storage in said second reservoir 140, where it can also be mixed with the additives. Downstream of the process station 200, the resulting second permeate E is conveyed via the return line 120 into the first reservoir 100 - which
20 therefore acts also as a storage facility for the processed wine, i.e. the final product of the process according to the present invention - whereas the resulting second concentrate F is conveyed back via the connection line 130 into the second reservoir 140.

The data set forth in the attached Table summarize the results of
25 three wine deacidification test runs, the first one of which was performed without the use of any additive (and therefore without any intermediate step between the two separation stages), while the remaining two ones have been carried out under utilization of two different additives between the two separation stages to process the first permeate C.

30 As shown by these experimental data, a reduction in volatile acidity,

which goes to as far as 34% in test run no. 2, is in all cases reached, while it is only with the use of additives that the tartaric acid contents remains substantially unaltered.

For a correct understanding of the data displayed in the attached
5 Table, it is reminded that acetic acid falls into the category of volatile compounds that must be eliminated, while tartaric acid is an essential component of wine.

It shall finally be appreciated that, within the scope of the present invention as recited in the appended claims, the afore described process
10 and/or apparatus may be implemented according to other embodiments differing from the above-described ones.

ANNEX

Experimental results

Sample	Total acidity [g/l]	Volatile acidity [g/l]	Acetic acid [g/l]	Tartaric acid [g/l]
Test run no. 1 - Additive : none				
Initial wine (A)	7.38	2.00	1.45	1.07
First permeate (C)	0.70	0.56	0.56	0.40
Second permeate (E)	0.55	0.43	0.38	Traces
Reconstituted wine	7.12	1.85	1.45	0.80
Test run no. 2 - Additive (G) : sodium hydroxide				
Initial wine (A)	7.38	2.00	1.45	1.07
First permeate (C)	0.70	0.56	0.56	0.40
Second permeate (E)	0.00	0.03	0.03	0.41
Reconstituted wine	7.18	1.32	1.27	1.11
Test run no. 3 - Additive (G) : sodium bicarbonate				
Initial wine (A)	7.38	2.00	1.45	1.07
First permeate (C)	0.70	0.56	0.56	0.40
Second permeate (E)	0.00	0.43	0.08	0.41
Reconstituted wine	7.18	1.86	1.31	1.04

10

CLAIMS

1. Process for the deacidification of wine, i.e. for the reduction of the contents of volatile acid compounds that are present as a solution in said wine, **characterized in that** it comprises in a sequence:

15 - a first stage in which the solution to be processed (A) is separated into a first concentrate (B) and a first permeate (C), in which first permeate (C) there are included the compounds that are responsible for said volatile acidity of the wine, further to at least most of the solvent;

20 - a second stage in which said first permeate (C) is in turn separated into a second concentrate (F), in which there are included the compounds that are responsible for said volatile acidity of the wine, and a second permeate (E) that comprises the solvent.

25 2. Process according to claim 1, **characterized in that** it further comprises, between said first and said second stage, a third stage in which said second permeate (E) joins again said first concentrate (B) and is mixed up again with the initial solution (A).

30 3. Process according to claim 1 or 2, **characterized in that** it comprises, between said first and said second stage, an intermediate stage in which substances (G) are added to said first permeate (C) that are adapted to at least partially neutralize said compounds that are responsible for the volatile acidity of the wine.

4. Process according to claim 3, characterized in that said substances (G) that are added to said first permeate (C) are sodium, potassium or calcium compounds, such as in particular hydroxides, carbonates, tartrates or acetates.
5. Process according to any of the preceding claims, characterized in that during said first and said second stage of the process, the initial solution (A) and the first permeate (C), respectively, are held under pressure.
6. Process according to any of the preceding claims, characterized in 10 that it is carried out in a continuous manner, i.e. without any solution of continuity.
7. Process according to any of the preceding claims, characterized in that it is carried out in a discontinuous manner.
8. Apparatus for the deacidification of wine using to the process 15 according to any of the preceding claims, characterized in that said first stage is performed in an initial process station (3) in which *per se* known membrane means are used that are adapted to separate molecules with a size that is smaller than approximately 1,000 dalton.
9. Apparatus according to claim 8, characterized in that said second 20 stage is performed in a final process station (7) in which *per se* known membrane means are used that are adapted to separate molecules with a size of approximately 1,000 to 300,000 dalton.
10. Apparatus according to claim 9, characterized in that between said initial process station (6) and said final process station (7) it comprises a 25 reaction stage (6) to which solid or liquid additives (G) are added via an appropriate supply line (12).
11. Apparatus according to claim 10, characterized in that, within 30 said reaction stage (6), solid additives are filled into a vertical container (61) that is housed inside a tank (63) and has a bottom (62) in the form of a grille, or the like, in which said first permeate (C) is fed into the tank (63)

via a conduit (55) that terminates on top of said container (61), and then, upon undergoing the desired treatment imparted by the contact with said additives (G), flows through a siphon (64) housed within the same tank (63) prior to entering the conduit (56) that conveys it to said final process station 5 (7).

12. Apparatus according to any of the claims 8 to 11, characterized in that it comprises pump means (22, 26) adapted to keep the solution to be processed (A) under pressure as it is being treated in said initial process station (3).

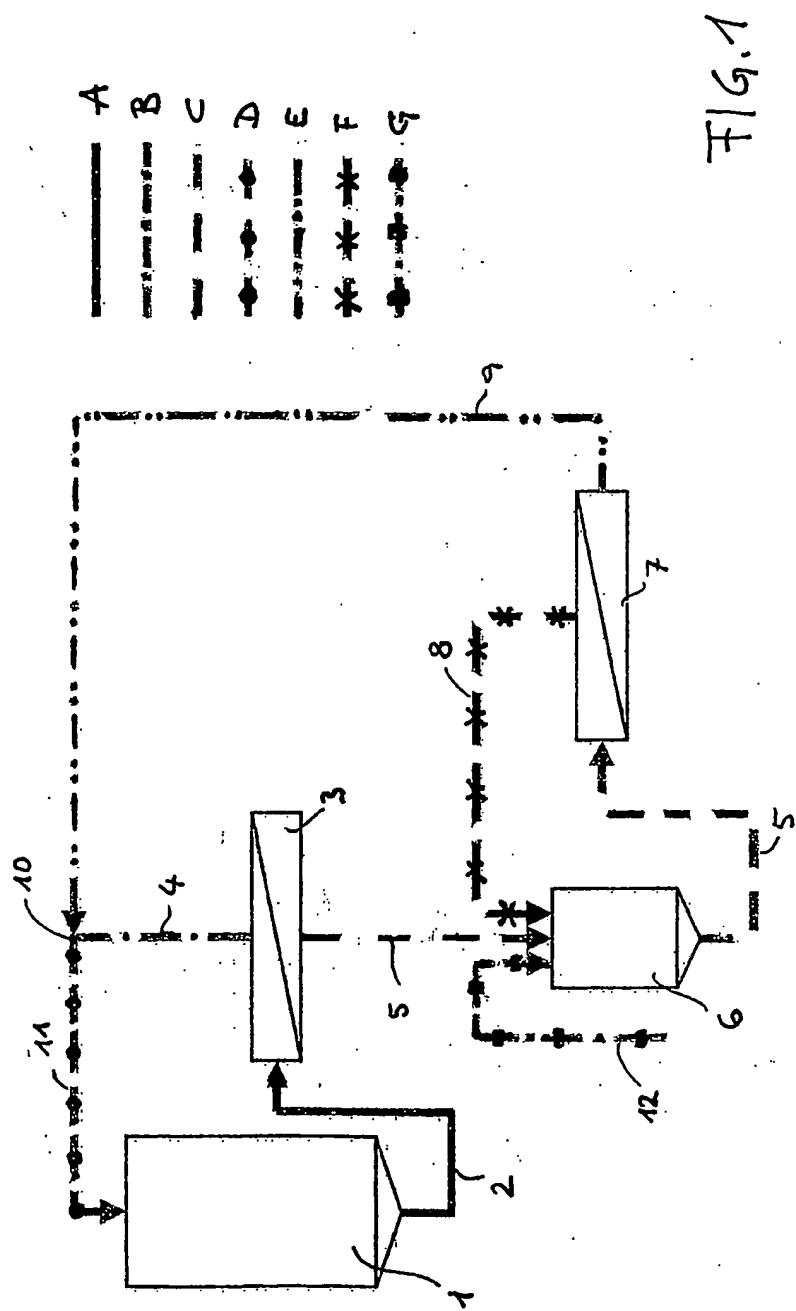
10 13. Apparatus according to any of the claims 8 to 12, characterized in that it comprises pump means (54) adapted to keep said first concentrate (C) under pressure as it is being treated in said final process station (7).

15 14. Apparatus according to any of the claims 8 to 13, characterized in that it comprises, ahead of said initial process station (3), filter means (25) that are adapted to prevent the membrane means that are a part of the two process stations (3, 7) to be damaged by solid particles that may be present in the solution to be processed (A).

15 15. Apparatus according to any of the claims 8 to 14, characterized in that it is a continuous apparatus.

20 16. Apparatus according to any of the claims 8 to 14, characterized in that it is a discontinuous apparatus in which a single process station (200) constitutes in a first stage said initial process station and, in a second stage, under possible utilization of filtering membranes provided with a different selectivity as compared with the ones used in said first stage, the 25 final process station, in which said apparatus further comprises: a first reservoir (100), which in the first stage collects and stores the solution to be processed (A) for delivery to said process station (200) and, in the second stage, receives the wine processed in the same process station; a second reservoir (140), which during said first stage receives the first permeate (C) 30 and, during said second stage, supplies said first permeate (C) to said

single process station (200).



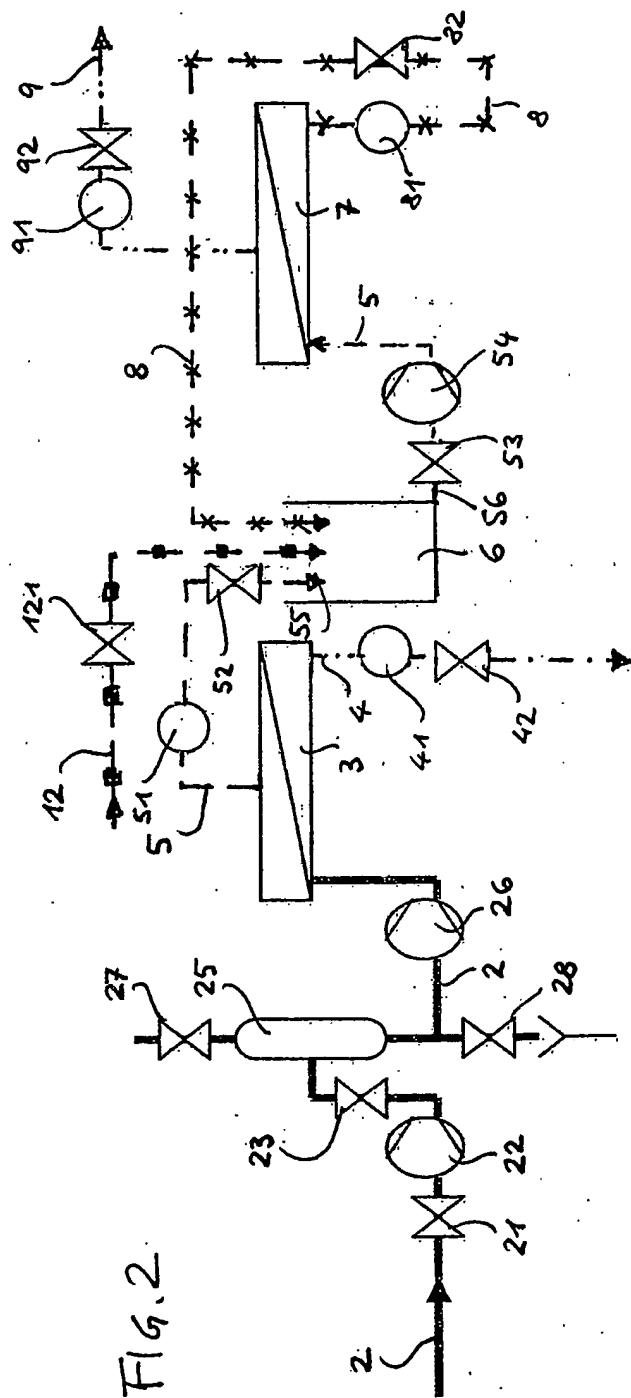
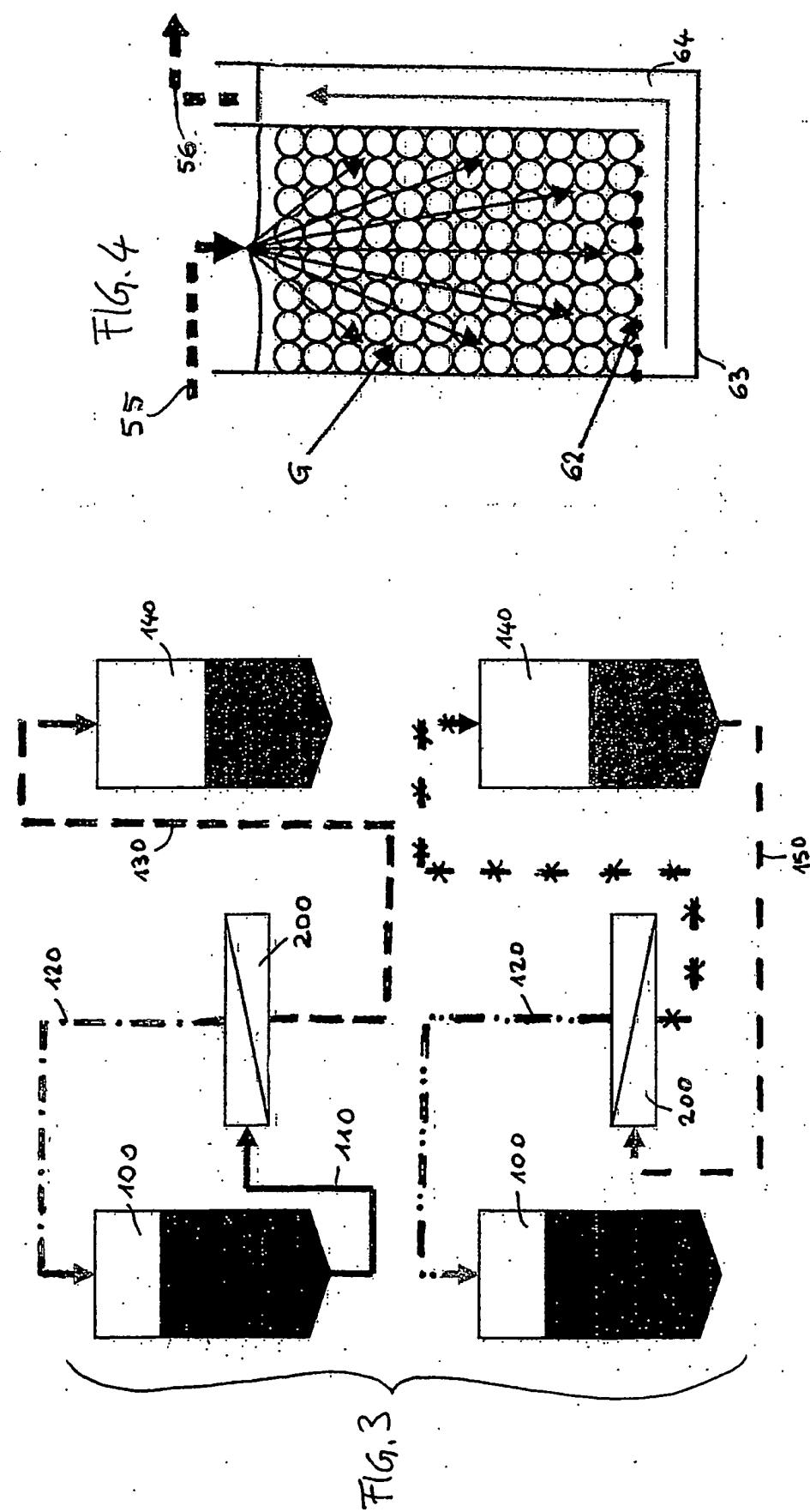


FIG. 2



INTERNATIONAL SEARCH REPORT

Int'l Application No
PCT/IT 02/00139

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C12G3/08 C12H1/14 C12H1/16 B01D61/58

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C12G C12H

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ, FSTA, BIOSIS, COMPENDEX

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 480 665 A (SMITH CLARK R) 2 January 1996 (1996-01-02) abstract	8,12-15
A	---	1-7,10, 11,16
X	US 4 499 117 A (BONNEAU MARC) 12 February 1985 (1985-02-12) claim 1; example 1	8,9, 13-15
A	---	1-7,10, 11,16
X	EP 0 460 339 A (WEIZMANN KIRYAT MEMBRANE PROD) 11 December 1991 (1991-12-11) example 7	8,9, 12-15
A	-----	1-7

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

* Special categories of cited documents :

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- *&* document member of the same patent family

Date of the actual completion of the international search

Date of mailing of the international search report

20 September 2002

07/10/2002

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Koch, J

INTERNATIONAL SEARCH REPORT

Information on patent family members

Inte
nal Application No
PCT/IT 02/00139

Patent document cited in search report		Publication date		Patent family member(s)	Publication date
US 5480665	A	02-01-1996		AT 171390 T AU 661912 B2 AU 4231993 A BG 62318 B1 BG 99165 A CA 2135305 A1 DE 69321229 D1 DE 69321229 T2 DK 639105 T3 EP 0639105 A1 ES 2123053 T3 HU 70805 A2 WO 9323151 A1 ZA 9303213 A	15-10-1998 10-08-1995 13-12-1993 31-08-1999 28-08-1995 25-11-1993 29-10-1998 18-02-1999 14-06-1999 22-02-1995 01-01-1999 28-11-1995 25-11-1993 14-06-1994
US 4499117	A	12-02-1985		FR 2497825 A1 AT 16199 T AU 8000782 A DE 3266969 D1 EP 0069137 A1 WO 8202405 A1	16-07-1982 15-11-1985 02-08-1982 28-11-1985 12-01-1983 22-07-1982
EP 0460339	A	11-12-1991		EP 0460339 A1 AU 624250 B2 AU 5706490 A	11-12-1991 04-06-1992 19-12-1991